Effect of boron addition on microstructure and mechanical properties of TiC/Ti6Al4V composites

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ABSTRACT

TiC/Ti6Al4V composites with different boron additions were successfully fabricated, and the effects of the boron content on the microstructure, mechanical properties of composites were investigated. The results show that the dendritic TiC in the composites is remarkably refined when the boron content is less than 0.06 wt.%. The average primary axial length of TiC dendrite is decreased from 150 μm to 50 μm according to the increasing of the boron content from 0.01 wt.% to 0.06 wt.%. And TiC dendrite gradually changes from the coarse dendrite to the fine dendrite or granular, but the abrasive property is obviously reduced with the increase of boron content. On the other hand, when the boron content further increases from 0.1 wt.% to 0.6 wt.%, the dendritic TiC can not be refined obviously, the fiber-shape TiB begins to appear and the abrasive property of composites is improved. The compressive strength and hardness (HRC) of composites are obviously increased in the whole addition range. The refinement mechanism of boron is attributed to the combined effects of the increase in nucleation rate at the constitutionally supercooled zone in front of the solid–liquid interface and the reduction in growth rate of TiC. The improvement of its abrasive property is mainly attributed to the existence of large size TiC and fiber-shape TiB during the abrasion.

1. Introduction

Ti6Al4V alloy has been widely used in artificial joint and tooth application because of its excellent mechanical properties and biocompatibility, and more its density near to the body bone [1,2]. However, when the implanted Ti6Al4V alloy was employed in wear conditions [3,4] there were large black debris around it, which displays the wear resistance property of the alloy should be improved more. Titanium alloy matrix composites reinforced with ceramics particles have significant potential in the field of biomedical materials due to their high hardness, excellent wear properties, good biocompatibility and corrosion resistance, and its close elastic modulus to human tissue [5–7].

Among the different fabricating techniques of TiC/Ti6Al4V composites, smelting–casting method is attractive since the TiC reinforcements fabricated by it are in situ and combined well with matrix [8–10]. But the size of the primary TiC dendrite was on a wide scale ranging from 1 μm to 100 μm and its stability was poor, which often resulted in the failure of the composites due to the fracture of TiC [11,12]. Therefore, controlling the shape and size of TiC in the titanium matrix composites is one of the keys to gain a high performance composite. Sarkara et al. [13] obtained considerable grain refinement after thermo-mechanical processing by adding boron and carbon in Ti-15–3β Ti alloy, and the refinement is more in carbon containing alloy because of finer and uniform distribution of TiC precipitates. By forging method Tjong and Ma [14] changed the dendritic TiC into fine particles to improve the properties of the composites. However, the addition cracks were also produced inside the TiC particles in this process. Ranganatha et al. [15] fabricated Ti–TiB–TiC composite using a processing method combining conventional melting and combustion synthesis reaction. They found the increase in strength of the rolled composite compared with the as-cast composite was attributed to the absence of dendritic structure. Lin et al. [16] studied the effect of cooling rate on the morphology and microstructure of TiC, and found that the cooling rate could modify the dendritic TiC and also affect the structure of TiC. But under a conventional casting condition, it is difficult to be realized. Wei et al. [17] investigated the effects of carbon sources, cooling rate, alloying elements and heat treatment on the morphology of TiC of TiC/Ti–6Al–4V composites. It was observed that different carbon sources of the composites led to different morphologies of TiC, and the carbon source of carbon powder was found to be more useful in restraining the dendritic growth of TiC. Increasing the cooling rate can reduce the size of TiC. They also found TiC particles became fine and dispersed when 0–3 wt.% Sn was added in the composites. And TiC particles in the

composites could be dissolved and gradually spheroidised with increasing holding time when heat treated at 1050 °C. Other researchers [18,19] also found by heat treatment, the morphology and size of TiC could be changed and the occurrence of cracks inside TiC particles could be prevented too. But if the growth of TiC crystal can be well controlled during the fabrication, the properties of composites will be improved significantly, and the process can also be simplified. The purpose of this paper is to evaluate the microstructure and properties of cast TiC/Ti6Al4V composites with various boron additions, and to analyze the effects of boron on TiC dendrite refinement.

2. Experimental procedure

TiC/Ti6Al4V composites with different boron additions were prepared by means of in situ melting–casting process. Firstly, High purity titanium powder (99.2%, 45 μm), aluminum powder (99.6%, 29 μm) and carbon powder (99.8%, <0.5 μm) were mixed according to designed ratios and ball-milled for 24 h. Then they were uniaxially pressed into a block with a compatibility of 50–60%, and then heated in a vacuum high temperature SHS reactive furnace to be synthesized TiC/Al master alloy. After that, according to the designed ratio, the TiC/Al master alloy, sponge titanium, vanadium power (99.9%, 20 μm), aluminum powder (99.6%, 29 μm) and boron power (98%, 45 μm) were melted in a consumable vacuum arc furnace equipped with a water-cooled copper crucible to prepare 15 vol.% TiC/Ti6Al4V composites. In order to ensure chemical homogeneity of the composites, electron magnetic agitation was used and the ingots were re-melted at least three times. The samples were obtained at the same radius range of each alloy ingot to ensure they have the same cooling rate. Boron contents of the composites were 0.01 wt.%, 0.02 wt.%, 0.04 wt.%, 0.06 wt.%, 0.1 wt.%, 0.2 wt.%, 0.4 wt.% and 0.6 wt.%, respectively. Phase identification was carried out via X-ray diffraction (XRD) using Rikagu D/max-RB diffractometer with Cu Kα radiation. The compression specimens were cut into flat plates from the composites ingots in a dimension of 5 mm × 5 mm × 10 mm, and then conducted at room temperature on CSS- 443- 00 universal machine. The rockwell hardness was measured under the testing load of 150 kg for 15 s. For microstructural observation, the samples were polished and etched in 2% HF + 8% HNO$_3$ + 90% H$_2$O solution. The morphologies of reinforcements were characterized using FEI Sirion scanning electron microscopy equipped with energy-dispersive X-ray spectrometer (EDS).

The wear tests were carried out under dry sliding and Hank's solution (NaCl 8.00 g L-1, KCl 0.40 g L-1, CaCl2 0.14 g L-1, NaHCO3 0.35 g L-1, MgSO4·7H2O 0.20 g L-1, Na2HPO4·12H2O 0.12 g L-1, KH2PO4 0.06 g L-1) lubrication condition at 37 °C ± 0.5 °C using a self-made wear testing machine, respectively. The rectangular TiC/Ti6Al4V composites specimens with a size of 10 mm × 10 mm × 3 mm were used for the investigation. The wear duality was a steel ball of GCr15 with HRC59 and the size was 6.35 mm in diameter. Wear tests were conducted at a rotating speed of 300 rpm using applied load of 4 N, and in each test the wear time was 30 min. The wear loss was calculated from the weight loss of the specimen weighed by an analysis balance with the sensitivity of ±0.01 mg. The worn surface was observed and analyzed using SEM.

3. Results and discussions

3.1. Phase identification

Fig. 1 shows X-ray diffraction patterns of TiC/Ti6Al4V composites with different boron contents. From the figure, we can see that when the addition content of boron is less than 0.06 wt.%, the phases mainly consists of α-Ti and TiC. It shows that no new phase
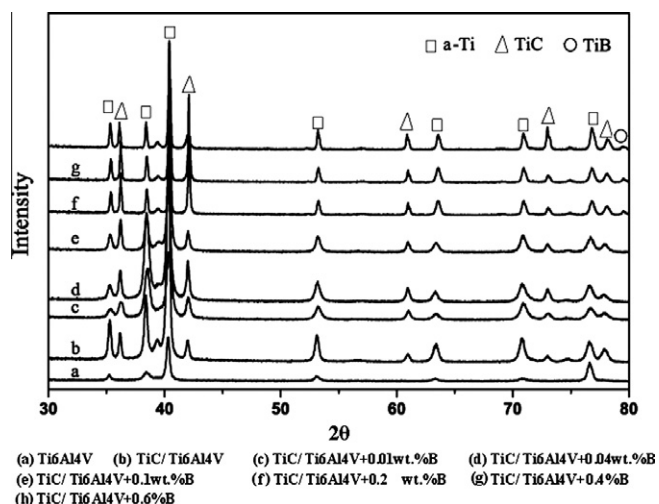

Fig. 1. XRD patterns of the 15 vol.% TiC/TI6A14V composites with different boron content.

formed in this range of boron addition. But when the addition of boron reaches 0.2 wt.%, TiB phase begin to appear in the composites.

3.2. Effect of boron addition on TiC morphology

The microstructure of 15 vol.% TiC/Ti6Al4V composite is shown in Fig. 2. It shows that TiC phases are distributed uniformly in the titanium alloy matrix. The primary dendrite is large and its average primary axial length is about 150 μm. TiC phase with large dendrite will lead to poor stability of the composites, and it often results in the failure of the composites because of the fracture of TiC [2,14,20].

The microstructures of TiC/Ti6Al4V composites with different boron contents are shown in Fig. 3. In the composite with 0.01 wt.% boron, the average primary axial length of dendrite TiC is approximate 100 μm, as shown in Fig. 3a, and it is obviously refined in comparison with that in the composites without boron addition. The distribution of TiC is uniform. In the composites with 0.04 wt.% and 0.06 wt.% boron content, as shown in Fig. 3c and d, the average primary axial length of the dendrite TiC decreases to about 50 μm, and some short-bar shape or particulate TiC phases appear. When the content of boron exceeds 0.1 wt.%, TiB begin to form by the reaction between the super-saturation boron and Ti. TiB mainly
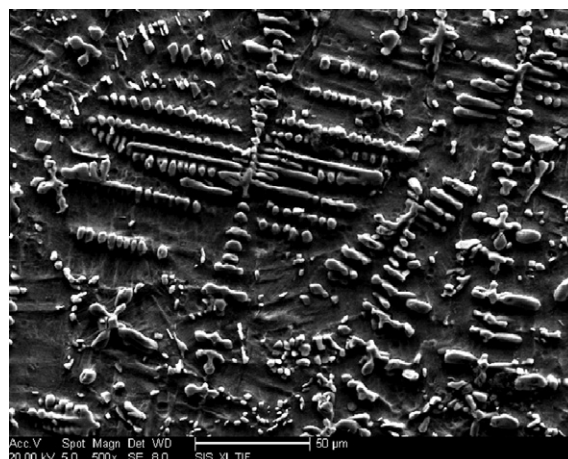

Fig. 2. Microstructure of 15 vol.% TiC/TI6A14V composites.

(a) 0.01 wt. %B   (b) 0.02 wt.%B   (c) 0.04 wt.%B   (d) 0.06 wt.%B
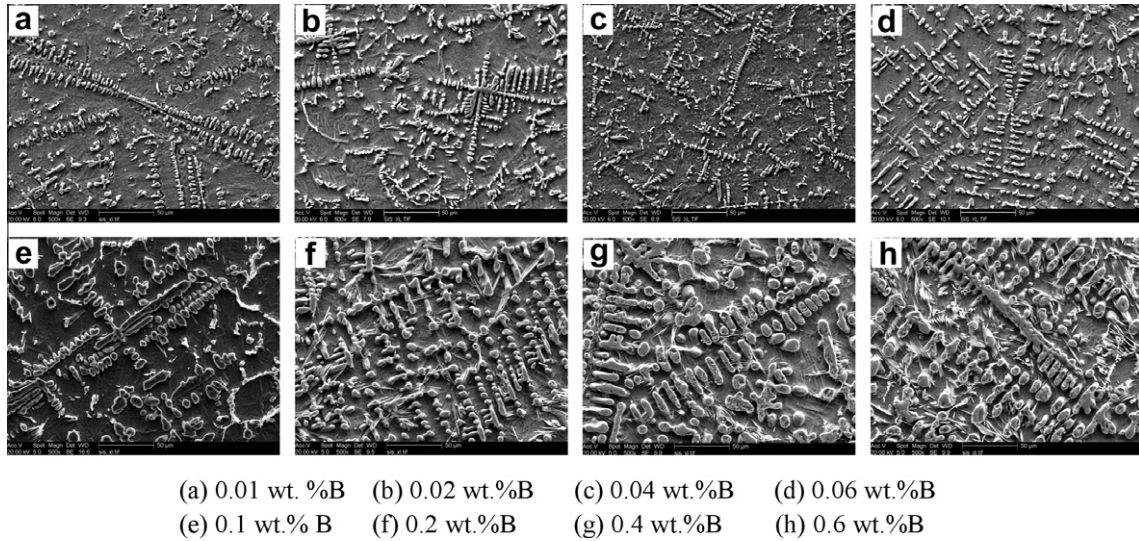(e) 0.1 wt.% B   (f) 0.2 wt.%B   (g) 0.4 wt.%B   (h) 0.6 wt.%B

Fig. 3. The effect of boron content on the morphology of TiC in TiC/Ti6Al4V composites.

scattered around TiC during the solidification process. With the further increasing of the boron content, the amount of TiB increases gradually, and the size becomes larger, while the morphology and the distribution of dendritic TiC have no significant changes.

The crystal growth is normally influenced by the mass transfer, the solute distribution and the interface energy at the liquid–solid interface. Also the absorbed state of the solute element on the crystal surface can be changed by adjusting the chemical composition of the alloy. Therefore, the crystal growth condition and growth manner can also be controlled, which will result into the changes of the crystal morphology [21]. According to the Ti–C binary phase diagram [22],the eutectic reaction occurs at 1650 °C in the Ti-rich corner with a carbon content of about 0.5 wt.%. And there is peritectic reaction in the Ti-rich side in the Ti–Al phase diagram. Therefore, it is a eutectic–peritectic ternary phase diagram for Ti–Al–C system in Ti-rich corner. When the TiC content is 15 vol.%, the composites are in a two-phase region of TiC and liquid phase in initial solidification. Decreasing with the temperature, the composites are in a two-phase region of TiC and β-Ti, and then in a two-phase region of TiC and α-Ti.

The crystal of TiC is a face center cubic (fcc) structure. According to Jackson's theory that the interface of equilibrium structure has the lowest free energy and the criterion of interface structure [23]:

$$a = \frac{\Delta H^0}{KT_m} \cdot \frac{\eta}{\gamma} \tag{1}$$

where ΔH^0 is the latent heat of crystallization (J/atom), K is Boltzmann's constant, T_m is the melting point, η is the coordination number in the surface layer, and γ is the coordination number for one atom in the solid state. When $a \leqslant 2$, the solid–liquid interface exhibits rough interface and its growth model is mainly continuous growth. From the literature [24], the maximum value of the parameter a of TiC is 1.87 and below 2, so the solid–liquid interface of TiC is rough and it grows freely in a continuous growth way. The composite was solidified in water-cooled copper crucibles and the cooling speed was high, so the under-cooling degree at the solid–liquid interface front was high. When the primary TiC grows into the super-cooled melt, a small destabilization in the solid–liquid interface front will induce the small projecting portion which can attain more precipitation potential and continue to grow into the liquid alloy. And the secondary and third dendrite arms can grow further at both sides of the projecting portion. Thus the morphology of the primary TiC is usually dendrite [25].

TiC refinement is based on nucleation in the constitutionally super-cooled region in front of the solid–liquid interface. According to the constitutional super-cooling theory, the formation of dendrites in alloys is often attributed to constitutional super-cooling whereby solute is rejected into the melt as alloy solidifies and a solute-rich layer can be built-up in front of the solidification interface. For alloys which contain boron, solute enrichment of boron is built up in the liquid in front of the solid–liquid interface because the solid solubility of boron in titanium is below 0.02 wt.% [26]. This solute enrichment causes a corresponding variation in the liquidus temperature leading to higher constitutional super-cooling. Thus the melt contains a high concentration of boron; more boron solute would be rich in front of the solid–liquid interface due to the rejection as the alloy solidifies. The constitutional super-cooling in front of the solid–liquid interface could, therefore, be very significant. The morphology of the dendrictic TiC depends on the extent of the constitutional super-cooling and the level of boron addition. Tamirisakandala et al. [27] suggested that under the condition of extreme constitutional super-cooling, nucleation could take place in the liquid in front of the solid–liquid interface. Hence, excess boron will be rejected from TiC nuclei into the melt as the composites solidifies and a solute-rich layer will be built-up in front of the solidification interface resulting in constitutional super-cooling, which in turn provides an additional driving force for the nucleation of fine TiC. Furthermore, the barrier formed by excess boron in the solid–liquid interface reduces the growth rate of TiC. The combined effects of increase in nucleation rate and reduction in growth rate yield smaller TiC. Boron is active at high temperature and can be easily adsorbed on the surface of TiC. This adsorption hinders the growth of TiC, but also makes the more constitutional super-cooling form in front of the solid–liquid interface, which accelerates the nucleation of TiC. Meanwhile, the enrichment of boron on the surface of TiC makes TiC ramify and the finer pinch form which makes the branch blow out to fine short bar shape or even particle shape. But when the content of boron increases to a certain extent, TiB will be formed by the reaction between boron and Ti in the composites, so the refining effect of boron on TiC is reduced or even lost.

3.3. Effect of boron addition on mechanical properties

Fig. 4 illustrates the hardness of the composites with different boron contents. It can be seen that the addition of boron enhances

the hardness of the composites. With the increasing of boron content, the increasing tendency of the hardness is very evident when boron content is less than 0.06 wt.% and it becomes slowly when boron content is more than 0.06 wt.%. This can be explained by the state of the hard phase in the depression which was produced in the hardness test. When the boron content is less than 0.06 wt.%, with the increasing of boron content, the size of TiC in the composites becomes smaller (as sketched in Fig. 3a–d, this brings about the sharply increasing of the quantity of hard phase TiC in the impress and it results into the evidently increasing of the hardness. When the boron content exceeds 0.06 wt.%, the size and the quantity of the hard phase TiC changes little, and with the increasing of boron content, the quantity of the hard phase TiB becomes more, but the increasing quantity of TiB is small, so the tendency of the hardness is also up, but the increasing slope becomes slowly.

Fig. 5 is the curve of compressive strength of TiC/Ti6Al4V composites varied with the boron content. It can be concluded that the addition of boron enhances the compressive properties of the composites. With the increasing of the boron content, the compressive strength of the composites also increases. As we know, with the increasing of the boron content from 0.00 wt.% to 0.06 wt.%, TiC dendrite gradually changes from the coarse dendrite to the fine dendrite or granular, and the refinement of TiC is beneficial to raising compressive properties of composites. Lu et al. [28] reported the mechanical properties of TiC/Ti composites improve significantly due to the incorporation of reinforcement. The addition of Al improves the mechanical properties of the composites by refining the reinforcement and matrix alloy. They considered the strengthening mechanisms mainly include the following factors: (a) bearing load by the TiC particles, (b) refinement of the grain size, and (c) intrinsic strengthening of the matrix alloy by
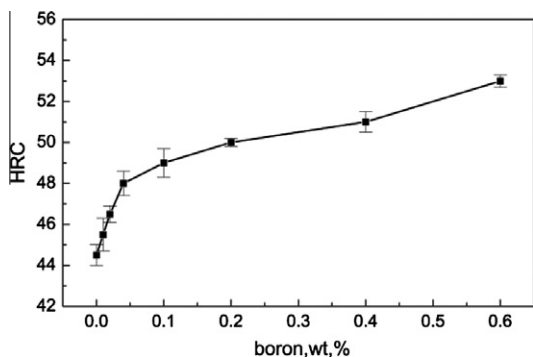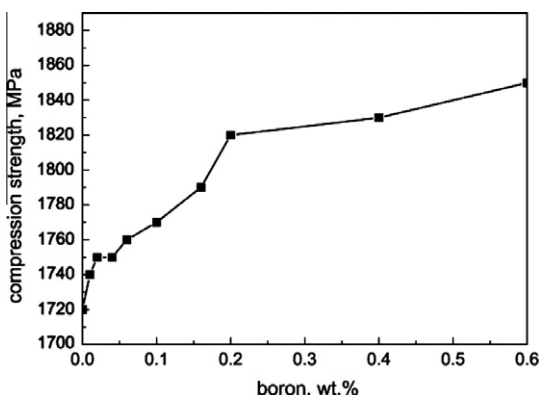
high-density dislocations. When a dislocation passes into the near grains or dendrites of different orientation, it has to change its moving direction. The hard TiC in the composites acts as obstacles to the motion of dislocations. Meanwhile, the atomic disorder within a boundary region will result in a discontinuity of slip planes from one to the other. Consequently, the strengthening mechanism of the composites may be mainly attributed to the load transfer from the soft matrix onto the hard reinforcements as well as dispersion strengthening from the finer reinforcements. When the addition content of boron reaches 0.1 wt.%, another phase TiB begins to form, which also contributes to the compressive strength of the composites. The mechanical properties of Ti matrix composites have been improved at large scale due to the following reasons: TiB whiskers and TiC particles undertaking load, refining of titanium matrix alloy grains and high density dislocation in Ti matrix alloy [29,30].

3.4. Effect of boron addition on abrasive property

3.4.1. Wear rate

Fig. 6 is the effect of boron addition content on the wear rate of TiC/Ti6Al4V composites. It can be seen whether it was under dry or Hank's solution lubrication wear condition, the wear rate shows an increasing tendency with the increasing of boron content when the addition content of boron is less than 0.06 wt.%. But when the boron content exceeds 0.06 wt.%, the wear rate decreases, which indicates that the abrasive property of the composites was improved. In the presence of lubricant, the wear rate is only 1/3 of that under dry wear condition.

In general, the wear resistance of materials will improve with the increase of hardness, but from Figs. 4 and 6, with the increasing of boron content from 0.00 wt.% to 0.06 wt.%, the increase tendency of the wear resistance is not according to the tendency of the hardness. The wear resistance is relevant not only to its strength and hardness, but also to its structure of the wear surface. The relationship between hardness and abrasion resistance is not simple direct ratio [31]. When the addition content of boron is less than 0.06 wt.%, TiC dentrite is refined by the addition of boron, and the site of the TiC particles in the matrix in the wear surface is shallow, so they can be easily peeled with the matrix by the furrow cut of the friction pairs and form into the abrasive dust. The abrasive dust may be adhered onto the wear duality, so the wear between the original friction pairs and the composites changes into the wear of the composite duality to composite to some extent. This results into the high wear rate and leads to the decrease of the wear resistant property of the composites. And the finer the TiC is, the more obvious this action is. From Fig. 3, it can be seen that when the boron content is in the range from 0.01 wt.% to 0.06 wt.%, the size of TiC becomes smaller with the increasing of the boron content gradually, so that the

Fig. 4. Effect of boron content on the hardness of TiC/Ti6A14V composites.

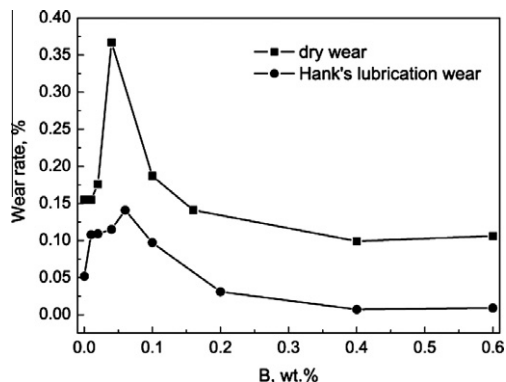Fig. 5. The compressive strength value of the composites varied with boron content.

Fig. 6. Effect of boron content on wear rate.

abrasive property of the composites decreases gradually. When the boron content continues to increase, the abrasive property of the composites begins to increase. The reasons might be due to that the morphology of TiC is almost not affected by the addition of boron (as sketched in Fig. 3e–h). Now the size of the TiC is larger, and its depth in the matrix increases, so the protection for the matrix from the furrow cut by the friction pairs is strengthen, that improves the abrasive property. In addition, a few fibrous TiB was generated around TiC, and they were also employed in the wear, which is equivalent to increase the wear resistant phase, so the abrasive property of the composites is improved. Yang et al. [32] reported that the hard particles will inhibit the plastic deformation and fracture of the soft matrix and act as load bearing components. This effect greatly reduces the direct load on the soft matrix and the composites are expected to have smaller accumulative strain and strain energy on the contact surface during sliding, which impedes the adhesion behavior between the sample and the steel ball and leads to a smaller wear rate. They also reported the pinning effect of TiB2 and TiC particles can effectively reduce the wear rate and friction coefficient and enhance the wear resistance of the composites.

3.4.2. Worn surface morphology under dry sliding condition

The worn surface morphologies of TiC reinforced Ti6Al4V composites under dry sliding condition are shown in Fig. 7. It can be seen from the figure when the boron content is less than 0.06 wt.%, there are some larger cutting and furrow marks and large adhesive deformation, termed as adhesive wear, on the worn surface. Under a high applied load, plastic flow is produced on the wear specimen, which results in the accumulating of the dislocation and leads to the microcracks in the later. When the cracks is expanded to a certain size, the worn debris is formed and flakes off the worn surface. From the observation of the morphology of the composites wear debris, it can be seen that when the addition of boron increases from 0.01 wt.% to 0.06 wt.%, the shape of the wear debris varies gradually from a small powder to a larger block, which indicates that the wear level increases with the increase of the addition content of boron.

When the boron content is more than 0.1 wt.%, the new-formed TiB phase is also employed in the wear, which results into the increasing wear resistance of the composites. Therefore, the cuttings and the furrow traces on the grinding surface are gradually weakened, and the intermittent wear trace increases.

3.4.3. Worn surface morphology under Hank's solution lubrication condition

The effects of the boron content on the wear surface morphology of the composites under the condition of Hank's solution lubrication are shown in Fig. 8. The change regularity of surface morphology is almost the same as that under the dry wear. When the boron content is 0.06 wt.%, a large number of peeling layer and
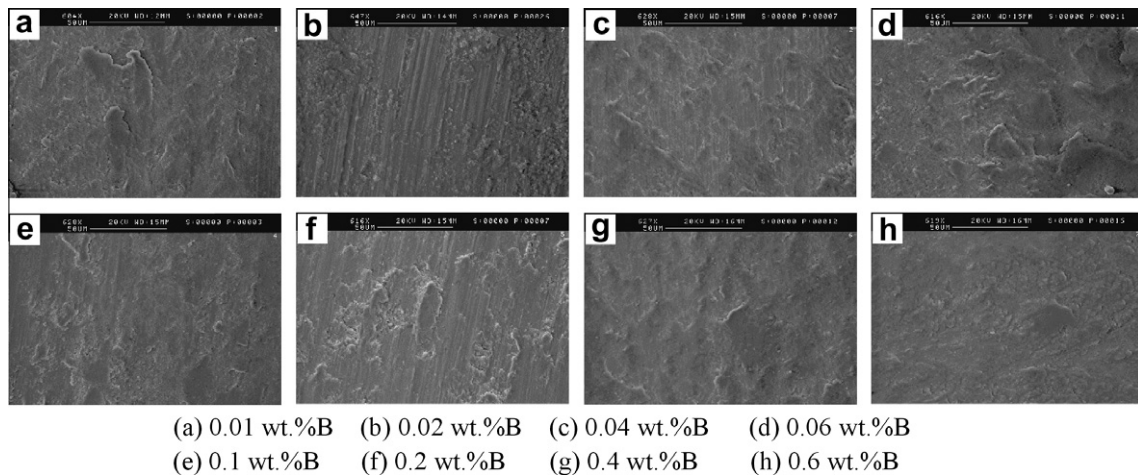

(a) 0.01 wt.%B   (b) 0.02 wt.%B   (c) 0.04 wt.%B   (d) 0.06 wt.%B
(e) 0.1 wt.%B   (f) 0.2 wt.%B   (g) 0.4 wt.%B   (h) 0.6 wt.%B

Fig. 7. Effect of boron content on the dry wear surface morphology of TiC/Ti6A14V composites.

(a) 0.01wt.%B   (b) 0.02wt%B   (c) 0.04wt.%B   (d) 0.06wt.%B
(e) 0.1wt.%B   (f) 0.2wt.%B   (g) 0.4wt%B   (h) 0.6wt%B
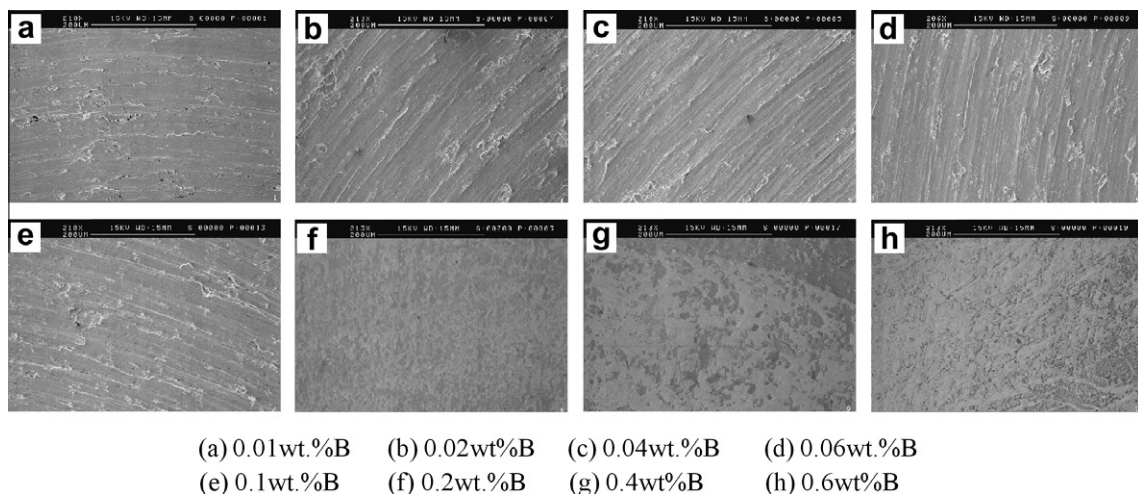
Fig. 8. Effect of boron content on Hank's lubrication surface morphology of TiC/Ti6A14V composites.

furrows are on the wear surface, which shows that the damage is very serious. With the boron content increases, the TiB reinforced phase appears, and under the condition of Hank's solution as lubricant, the wear surface is gradually smooth and there is few grinding marks and only some slight peeling layer on it (shown in Fig. 8e–h).

4. Conclusions

TiC/Ti6Al4V composites with different boron additions were successfully fabricated by means of in situ melting–casting process. TiC dendrite in TiC/Ti6Al4V composites is obviously refined by adding a little boron. With the addition of 0.06 wt.%, the average primary axial length of TiC dendrite decreases to 50 μm from 150 μm, and short-bar shape and particles TiC also begins to appear. However, further addition of boron can not refine TiC dendrite anymore and some TiB begin to form. The refinement of TiC is attributed to the combined effects of the increase in nucleation rate and the reduction in the growth rate of TiC induced by boron addition. With the increasing of the boron content in the whole addition range, the compressive strength and the hardness of the TiC reinforced Ti6Al4V composites increase. When the boron content is in the range of 0.01–0.06 wt.%, the wear resistance of TiC/Ti6Al4V composites decreases with the increase of boron content. While the wear resistance of the composites is significantly improved when the added boron is more than 0.1 wt.%, which is mainly due to the existence of large size TiC and fiber-shape TiB.

Acknowledgements

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